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IS 14305 (1995): Basic lead carbonate [CHD 1: Inorganic Chemicals]

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क्षारीय सीसा कार्बोनेट — विशिष्टि

Indian Standard

BASIC LEAD CARBONATE — SPECIFICATION

UDC 661·852·622·8

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1995

Price Group 3

General Inorganic Chemicals Sectional Committee, CHD 003

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Basic lead carbonate is also known as lead sub-carbonate and white lead. It is a white amorphous powder, insoluble in water but soluble in acids.

Basic lead carbonate is used as a pigment in industrial paints, in putty and ceramic glazes.

The composition of the committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

BASIC LEAD CARBONATE — SPECIFICATION

1 SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for basic lead carbonate.

2 REFERENCES

2.1 The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
33 : 1992	Inorganic pigments and extenders for paints — Methods of sampling and test (*third revision*)
264 : 1976	Nitric acid (*second revision*)
266 : 1993	Sulphuric acid — Specification (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of dry powder or in such a condition that it can be reduced to the powder form by crushing, without grinding action, under a palette knife.

3.2 Composition

The material, dried as described under **8** of IS 33 : 1992, shall consist of not less than 99 percent by mass of basic carbonate of lead which, in turn, shall consist of not less than 20 percent by mass and not more than 33 percent by mass of lead hydroxide chemically combined with not more than 80 percent by mass and not less than 67 percent by mass of normal carbonate of lead.

3.2.1 The composition of the material shall be determined as given in Annex A.

3.3 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in IS 33 : 1992. Reference to the relevant clauses of IS 33 : 1992 is given in col 4 of the table.

4 PACKING AND MARKING

4.1 Packing

The material shall be supplied packed in suitable containers as agreed to between the purchaser and the supplier.

Table 1 Requirements for Basic Lead Carbonate

(*Clause* 3.3)

Sl No.	**Characteristic**	**Requirements**	**Methods of Test (Ref to Cl No. in IS 33 : 1992)**
(1)	(2)	(3)	(4)
i)	Volatile matter, percent by mass, *Max*	0·5	8
ii)	Residue on sieve, percent by mass, *Max*	0·3	9
iii)	Oil absorption	7 to 12	10
iv)	Colour	Close match to the approved sample	11
v)	Reducing power	Not inferior to the approved sample	15
vi)	Matter soluble in water, percent by mass, *Max*	0·5	19
vii)	Alkalinity, percent by mass, *Max*	0·1	20

4.2 Marking

Each package shall be legibly and indelibly marked with the following information:

a) Name of the material;

b) Indication of the source of manufacture;

c) Gross and net mass of the material;

d) Batch number or lot number in code or otherwise; and

e) Month and year of manufacture.

4.2.1 The material is poisonous in nature. This fact should be clearly and boldly marked on the packages.

4.3 BIS Certification Marking

4.3.1 The packages may also be marked with the Standard Mark.

4.3.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples of the material, number of tests to be performed and the method of determining the criteria of conformity of the material to the requirements of this specification shall be as prescribed under **5** of IS 33 : 1992.

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 3.2.1)

ANALYSIS OF BASIC LEAD CARBONATE

A-0 GENERAL

A-0.1 Two methods are prescribed for estimation of basic lead carbonate (white lead). The volumetric method as prescribed in **A-1** shall be used for routine analysis and the gravimetric method prescribed in **A-2** shall be used as the referee method in case of any dispute.

A-1 VOLUMETRIC METHOD

A-1.1 Reagents

A-1.1.1 *Dilute Sulphuric Acid* — prepared by diluting 50 ml of concentrated sulphuric acid in a litre of water.

A-1.1.2 *Sodium Acetate Buffer Solution* (*p*H *5·3*) — prepared by dissolving 450 g of sodium acetate (AR grade) in 820 ml of deionised water, and adding to it 50 ml concentrated nitric acid S. G. 1·42 (AR Grade) and then mixing it thoroughly.

A-1.1.3 *0·05M EDTA Solution* — prepared by dissolving 40 g of di-sodium salt of ethylenediamine tetra-acetic acid in deionised water and diluting to 2 litre. This is standardized against pure lead nitrate (AR grade) or pure metallic lead.

A-1.1.4 *Xylenol Orange Indicator* (*0·5% Solution*) — prepared by dissolving 0·5 g of xylenol orange in 100 ml deionised water.

A-1.1.5 *Standard Lead Nitrate Solution* (*0·05M*) — prepared by dissolving 8·28 g accurately weighed AR lead nitrate in 200 ml deionised water. Add 1 ml nitric acid (sp gr 1·42) to it and then dilute to 500 ml in a volumetric flask with deionised water.

A-1.1.6 *Deionised Water*

A-1.2 Standardisation of EDTA Solution

Twenty five ml of standard lead nitrate solution (**A-1.1.5**) is taken in a 250-ml conical flask. Add 40 ml sodium acetate buffer solution to it and then dilute it to 150 ml by deionised water. Add 6 drops xylenol orange solution and titrate with EDTA solution (**A-1.1.3**) until the colour changes from the original reddish purple to a clear yellow.

A-1.3 Procedure

Weigh accurately about 0·3 g of the sample and transfer it to 250-ml beaker. Add 60 to 70 ml of dilute sulphuric acid and break up agglomerated particles by stirring well with a glass rod. Boil for 2 to 3 minutes taking care to prevent bumping and wash down the sides of the beaker with about 20 to 30 ml of water. Cool the beaker in ice cold water for one hour (or allow it to stand overnight). Then filter the residue through sintered glass crucible (No. 4) or Gooch crucible. Transfer the residue to the crucible and wash it with 10 ml of cold water twice. Keep this residue under slow suction with hot sodium acetate buffer solution (5 times) taking 8 ml each time. Wash with deionised water 2 to 3 times. Transfer the combined filtrate into the same beaker and make the volume up to 150 ml with deionised water and boil the solution until no white particles of $PbSO_4$ remains. Cool it under tap. Add 6 drops xylenol orange indicator and titrate with EDTA until the colour changes from the original reddish purple to a clear yellow.

A-1.4 Calculation

$$\text{Total lead content (expressed as PbO), percent by mass} = \frac{V_2 \times 27{\cdot}875}{V_1 \times W}$$

where

V_1 = volume in ml of EDTA solution required for 25 ml standard lead nitrate solution,

V_2 = volume in ml of standard EDTA solution required for the pigment, and

W = mass in g of the pigment taken for analysis.

NOTES

1 For routine analysis PbO content in the sample (provided no other metallic impurities are present) may be determined directly by dissolving the accurately weighed sample in sodium acetate buffer solution and then titrating the cold solution with standard EDTA using xylenol orange as indicator (that is, by-passing Sulphuric Acid Treatment step).

This procedure is very fast and one can do a number of samples at a time within 1 hour of time.

2 For routine analysis, if the PbO content of the sample is between 85·3 and 86 percent, estimation of carbon dioxide (*see* **A-2.5**) may not be carried out.

A-2 GRAVIMETRIC METHOD

A-2.1 Reagents

A-2.1.1 *Dilute Hydrochloric Acid* — 1 : 1.

A-2.1.2 *Hydrogen Sulphide* — gas.

A-2.1.3 *Sulphuric Acid* — relative density 1·84 (*see* IS 266 : 1993).

A-2.1.4 *Ethyl Alcohol* — 90 percent (*v*/*v*).

A-2.1.5 *Nitric Acid* — relative density 1·14 (*see* IS 264 : 1976).

A-2.1.6 *Ammonia Solution* — 1 : 1.

A-2.1.7 *Dilute Sulphuric Acid* — 3 percent (*v*/*v*).

A-2.1.8 *Hydrogen Sulphide Solution* — saturated.

A-2.2 Procedure

Weigh out accurately 0·5 to 1 g of the pigment in a dry 400-ml beaker. Add about 60 ml of dilute hydrochloric acid, cover with a clock-glass and boil slowly over a wire gauge for about 5 to 10 minutes. Dilute to about 200 ml with hot water, filter and wash the residue, if any, on the filter paper (Whatman No. 30) with hot water until a few drops of the filtrate do not give test for lead with hydrogen sulphide solution. Nearly neutralize the filtrate with ammonia solution in presence of a piece of litmus paper and add 5 ml of dilute hydrochloric acid. Make the volume of the solution to about 300 ml with water and pass hydrogen sulphide for about 15 minutes. Allow the precipitate to settle for 5 minutes and then filter on a filter paper (Whatman No. 30). Wash the precipitate by decantation once with hydrogen sulphide solution, transfer it to the filter and wash it again on the filter paper 4 to 5 times with hydrogen sulphide solution. Dissolve the precipitate in hot nitric acid, collect the solution in a 400-ml beaker and wash the filter paper 4 to 5 times with a jet of hot water, collecting the washings in the same beaker. Cool, wash down sides of the beaker with a small quantity of water and again evaporate to dense fumes of sulphur trioxide; cool, dilute carefully with 100 ml of water and add 100 ml of ethyl alcohol. Mix thoroughly and allow it to stand for at least 4 hours. Filter off the precipitate of lead sulphate through a weighed heat resistant crucible (Gooch or sintered glass No. 4) which has been tared after heating to 500°C. Transfer the precipitate to the filter by a jet of dilute sulphuric acid. Dry the crucible at 130°C in an oven for about half an hour. Transfer to a furnace maintained at 500°C for at least 30 minutes. Cool in a desiccator and weigh. Repeat the drying, cooling and weighing until constant mass is obtained.

A-2.3 Calculation

$$\text{Total lead content (expressed as PbO), percent by mass} = \frac{73{\cdot}6 \times M_2}{M_1}$$

where

M_1 = mass in g of the pigment taken for analysis, and

M_2 = mass in g of lead sulphate ($PbSO_4$) precipitate.

A-2.4 Normal lead carbonate equivalent of total lead (X) in the sample, expressed as percentage, is given by the formula:

$$X = \frac{88{\cdot}11 \times M_2}{M_1}$$

where

M_1 = mass in g of the sample, and

M_2 = mass in g of the lead sulphate.

A-2.5 Determination of Carbon Dioxide

A-2.5.1 *General*

The determination of lead carbonate content is derived from the determination of carbon dioxide (CO_2) content.

A-2.5.2 *Reagents*

A-2.5.2.1 *Nitric acid* — 4 N.

A-2.5.2.2 *Concentrated sulphuric acid* — relative density 1·84 (*see* IS 266 : 1993).

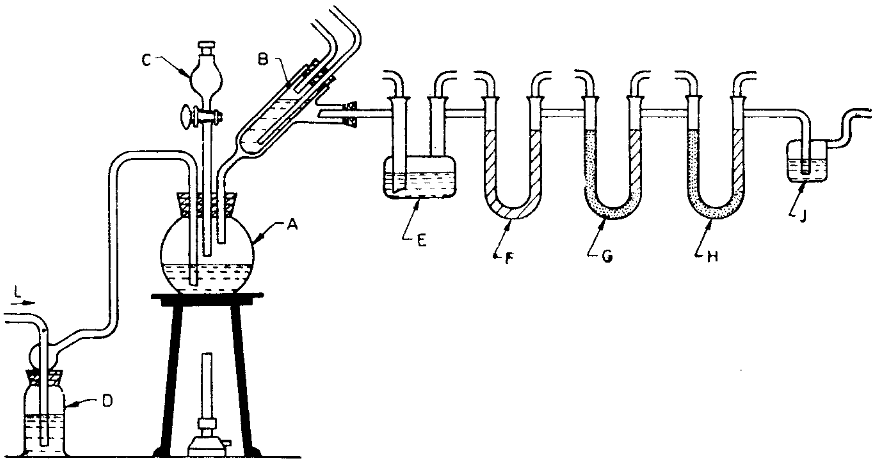
A-2.5.2.3 *Potassium hydroxide solution* — concentrated. Dissolve 50 g of potassium hydroxide in 100 ml of water.

A-2.5.2.4 *Magnesium perchlorate* — anhydrous.

A-2.5.2.5 *Indicating soda lime or soda asbestos.*

A-2.5.3 *Apparatus*

Use the apparatus for determination of carbon dioxide (CO_2) shown in Fig. 1. Before making a determination, prepare the apparatus by



A = boiling flask with round bottom and short neck, nominal capacity 250 ml
B = short reflux condenser
C = stoppered dropping funnel
D = wash-bottle containing potassium hydroxide solution
E = washing vessel containing concentrated sulphuric acid
F = U-tube containing a glass wool plug to trap spray and filled with anhydrous magnesium perchlorate
G and *H* = two ground-glass stoppered U-tubes filled with indicating soda lime or soda asbestos (size 10 to 14 mesh) in first two thirds of space, anhydrous magnesium perchlorate in the remaining space (at the side where the gas stream passes out)
J = indicating bubble counter containing concentrated sulphuric acid (alternatively a third U-tube filled with anhydrous magnesium perchlorate in first one-third of space and soda asbestos in the remaining two-thirds may be used)
L = stream of air.

NOTE — The absorption tubes *E* to *J* as well as the other components of the apparatus need not necessarily be of the form illustrated in the figure.

FIG. 1 APPARATUS FOR THE DETERMINATION OF THE PERCENTAGE OF CARBON DIOXIDE (CO_2)

slowly aspirating air and perform a blank test without any sample following the procedure given in A-2.5.4. The apparatus should be regarded as satisfactory only if less than 1 mg of carbon dioxide is obtained when the blank test is carried out.

A-2.5.4 *Procedure*

A-2.5.4.1 Weigh to the nearest 1 mg, 0·5 to 1·0 g of white lead into the flask *A*. Place 10 ml of water in the flask and assemble the apparatus as shown in Fig. 1. Turn the water on in the condenser *B* and draw air through the apparatus for 15 minutes. Close the taps in the absorption tubes *G* and *H* and remove the tubes. Wipe the tubes with a clean cloth and allow them to remain in the balance case for 30 minutes. Use a similar U-tube as a counterpoise and before weighing open all the taps momentarily to equalize the internal pressure. Reconnect the tubes to the apparatus after weighing. Pour 50 ml of nitric acid into the dropping funnel *C* and allow it to run gradually into the flask *A* until the funnel is almost empty.

A-2.5.4.2 When the reaction has subsided, heat the flask, regulating the heat to give a steady flow through the washing vessel *E*. Allow the contents of the flask to boil slowly, aspirating a slow stream of air for a few minutes. Remove the source of heat and draw air through the apparatus at the rate of two to three bubbles per second for a further 20 minutes. Then close the taps of the absorption tubes, disconnect the tubes and weigh them using the same procedure as for the first weighing.

A-2.5.5 *Calculation*

The content of lead carbonate ($PbCO_3$) of white lead *Y*, expressed as a percentage by mass, is given by the following formula:

$$Y = \frac{607{\cdot}4 \times M_3}{M_4}$$

where

M_3 = increase in mass in g of the absorption tubes, and

M_4 = mass in g of the test sample.

A-2.6 Calculation of Lead Hydroxide

Subtract the percentage of lead carbonate (Y) found in **A-2.5.5** from the percentage of lead carbonate equivalent of total lead (X) found in **A-2.4** and calculate the percentage of lead hydroxide [$Pb(OH)_2$] equivalent to the difference.

Percentage of $Pb(OH)_2$ (Z) is given by the following equation:

$$Z = 0.902\,7\,(X - Y)$$

A-2.7 Calculation of Percentage of Basic Carbonate of Lead (White Lead)

Percentage of basic carbonate of lead is equal to the sum of the percentage of lead carbonate (Y) found in **A-2.5.5** and percentage of lead hydroxide (Z) found in **A-2.6.**

Percentage of basic carbonate of lead $= Y + Z$.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

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(*Continued from page* 5)

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